Bis-μ-[1,3-bis(diphenylphosphino)propane]-dinitratodisilver(I)

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Abstract. $[Ag_2(NO_3)_2(C_{27}H_{26}P_2)_2]$, $[Ag(NO_3)\{Ph_2P(CH_2)_3PPh_2\}]_2$, $M_r = 1164\cdot6$, triclinic, $P\bar{1}$, $a = 11\cdot055$ (1), $b = 12\cdot838$ (1), $c = 10\cdot858$ (2) Å, $\alpha = 114\cdot05$ (1), $\beta = 112\cdot20$ (1), $\gamma = 90\cdot04$ (1)°, $V = 1281$ (1) Å^3, $Z = 1$ (dimer), $D_x = 1\cdot510$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot7107$ Å, $\mu = 0\cdot894$ mm^{-1}, $F(000) = 592$, $T = 293$ (1) K, $R = 0\cdot029$ for 3605 observed reflections. The title compound exists as a centrosymmetric dimer in which two Ag atoms are bridged by two $Ph_2P(CH_2)_3PPh_2$ ligands [Ag—P(1) 2·403 (1), Ag—P(2′) 2·426 (1) Å; P(1)—Ag—P(2′) 152·2 (1)°] such that a twelve-membered ring is formed. The coordination about each Ag atom is completed by a bidentate nitrate group [Ag—O(1) 2·581 (3), Ag—O(2) 2·674 (3) Å]. The nitrate groups are disposed above and below the ring.

Experimental. Air-stable $[Ag\{Ph_2P(CH_2)_3PPh_2\}NO_3]_2$ was isolated by slow evaporation of a chloroform solution containing equimolar quantities of AgNO$_3$ (Johnson Matthey) and $Ph_2P(CH_2)_3PPh_2$ (Strem). Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; ω:2θ scan technique. Cell parameters by least squares on 25 reflections ($11 \le \theta \le 15°$) (de Boer & Duisenberg, 1984) on a crystal 0·25 × 0·25 × 0·62 mm. Analytical absorption correction applied; maximum and minimum transmission factors 0·805 and 0·601 (Sheldrick, 1976). Total of 5505 reflections ($1\cdot5 \le \theta \le 25°$) measured in the range $-13 \le h \le 13$, $-15 \le k \le 15$, $-12 \le l \le 3$. No significant variation in the net intensities of two reference reflections ($2\bar{2}4$, $5\bar{2}4$) measured every 7200 s. 4521 unique reflections ($R_{int} = 0\cdot013$) and 3605 satisfied $I \ge 2\cdot5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, and H atoms included at their calculated positions. At convergence $R = 0\cdot029$, $wR = 0\cdot034$, $w = [\sigma^2(F) + 0\cdot0025|F|^2]^{-1}$, $S = 1\cdot13$, $(\Delta/\sigma)_{max} \le 0\cdot01$, $\Delta\rho_{max} = 0\cdot53$, $\Delta\rho_{min} = -0\cdot83$ e Å^{-3}; no extinction correction applied. Scattering factors for neutral Ag corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149) and for

Table 1. *Fractional atomic coordinates and B_{eq} values*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq} (Å^2)
Ag	0·09749 (2)	0·19315 (2)	0·09666 (2)	3·65
P(1)	0·0766 (1)	0·1978 (1)	−0·1295 (1)	2·86
P(2)	−0·2265 (1)	−0·2231 (1)	−0·3492 (1)	2·76
C(1)	0·0165 (3)	0·0531 (2)	−0·2861 (3)	3·45
C(2)	−0·1077 (3)	−0·0101 (2)	−0·2984 (3)	3·03
C(3)	−0·1373 (3)	−0·1400 (2)	−0·4018 (3)	3·21
C(111)	0·2347 (3)	0·2408 (2)	−0·1280 (3)	2·92
C(112)	0·2658 (3)	0·3379 (3)	−0·1446 (3)	3·90
C(113)	0·3910 (4)	0·3670 (3)	−0·1348 (4)	4·66
C(114)	0·4848 (4)	0·3011 (4)	−0·1088 (4)	5·09
C(115)	0·4559 (4)	0·2021 (3)	−0·0929 (4)	4·72
C(116)	0·3310 (3)	0·1734 (3)	−0·1027 (4)	4·00
C(121)	−0·0350 (3)	0·2875 (3)	−0·1906 (3)	3·63
C(122)	−0·0573 (4)	0·2956 (4)	−0·3202 (5)	6·45
C(123)	−0·1423 (5)	0·3643 (6)	−0·3648 (8)	10·34
C(124)	−0·2053 (5)	0·4260 (5)	−0·2787 (8)	9·09
C(125)	−0·1858 (5)	0·4193 (4)	−0·1506 (7)	7·16
C(126)	−0·0996 (4)	0·3494 (3)	−0·1063 (5)	4·79
C(211)	−0·3849 (3)	−0·1754 (2)	−0·3805 (3)	3·25
C(212)	−0·4298 (4)	−0·1426 (3)	−0·2698 (4)	5·13
C(213)	−0·5517 (5)	−0·1066 (4)	−0·2925 (6)	7·29
C(214)	−0·6245 (4)	−0·1002 (4)	−0·4184 (6)	6·43
C(215)	−0·5808 (5)	−0·1321 (5)	−0·5280 (6)	6·54
C(216)	−0·4617 (4)	−0·1691 (4)	−0·5097 (4)	5·03
C(221)	−0·2655 (3)	−0·3702 (2)	−0·4961 (3)	3·06
C(222)	−0·1648 (3)	−0·4161 (3)	−0·5379 (3)	3·67
C(223)	−0·1917 (4)	−0·5289 (3)	−0·6463 (4)	4·60
C(224)	−0·3142 (4)	−0·5971 (3)	−0·7113 (4)	4·55
C(225)	−0·4131 (4)	−0·5527 (3)	−0·6695 (4)	4·64
C(226)	−0·3892 (3)	−0·4392 (3)	−0·5624 (4)	4·00
N(1)	−0·1697 (3)	0·1294 (3)	0·0938 (4)	5·22
O(1)	−0·1085 (3)	0·0580 (2)	0·0410 (3)	5·18
O(2)	−0·1234 (5)	0·2324 (3)	0·1414 (6)	12·24
O(3)	−0·2689 (4)	0·0985 (3)	0·1038 (5)	11·01

Table 2. *Selected interatomic distances (Å) and bond angles (°)*

Primed atoms are related by a centre of inversion.

Ag—P(1)	2·403 (1)	Ag—P(2′)	2·426 (1)
Ag—O(1)	2·581 (3)	Ag—O(2)	2·674 (3)
P(1)—C(1)	1·831 (3)	P(1)—C(111)	1·824 (3)
P(1)—C(121)	1·814 (3)	P(2)—C(3)	1·833 (3)
P(2)—C(211)	1·810 (3)	P(2)—C(221)	1·823 (3)
C(1)—C(2)	1·528 (4)	C(2)—C(3)	1·531 (4)
N(1)—O(1)	1·221 (3)	N(1)—O(2)	1·233 (4)
N(1)—O(3)	1·224 (4)		
P(1)—Ag—P(2′)	152·2 (1)	P(1)—Ag—O(1)	108·6 (1)
P(1)—Ag—O(2)	111·2 (1)	P(2′)—Ag—O(1)	97·8 (1)
P(2′)—Ag—O(2)	111·2 (1)	O(1)—Ag—O(2)	46·9 (1)
Ag—P(1)—C(1)	111·8 (1)	Ag—P(1)—C(111)	113·7 (1)
Ag—P(1)—C(121)	116·6 (1)	C(1)—P(1)—C(111)	101·9 (1)
C(1)—P(1)—C(2)	104·9 (1)	C(111)—P(1)—C(121)	106·6 (1)
Ag′—P(2)—C(3)	110·6 (1)	Ag′—P(2)—C(211)	114·6 (1)
Ag′—P(2)—C(221)	117·6 (1)	C(3)—P(2)—C(211)	104·7 (1)
C(3)—P(2)—C(221)	103·4 (1)	C(211)—P(2)—C(221)	104·7 (1)
P(1)—C(1)—C(2)	112·4 (2)	P(2)—C(3)—C(2)	111·1 (2)
C(1)—C(2)—C(3)	112·3 (2)	Ag—O(1)—N(1)	100·5 (2)
Ag—O(2)—N(1)	95·5 (2)	O(1)—N(1)—O(2)	116·9 (3)
O(1)—N(1)—O(3)	120·5 (3)	O(2)—N(1)—O(3)	122·5 (3)

the remaining atoms as incorporated in *SHELX*76 (Sheldrick, 1976). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and numbering scheme used is shown in Fig. 1 [drawn with *ORTEP* (Johnson, 1971) at 15% probability levels].

Related literature. Dimeric ring structures similar to that observed in [Ag{Ph₂P(CH₂)₃PPh₂}NO₃]₂ are well known in Ag chemistry for compounds of the general formula [Ag(P—P)X]₂ where X is a uni-negative anion. Eight-membered rings have been characterized crystallographically in two bis(di-phenylphosphino)methane (dppm) complexes, *i.e.* [Ag(dppm)NO₃]₂ (Ho & Bau, 1983; Tiekink, 1990) and in the bis(dimethylphosphino)methane analogue [Ag(dmpm)NO₃]₂ (Karsch & Schubert, 1982).

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* Lists of structure factors, anisotropic thermal parameters, non-H atom bond lengths and angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53051 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
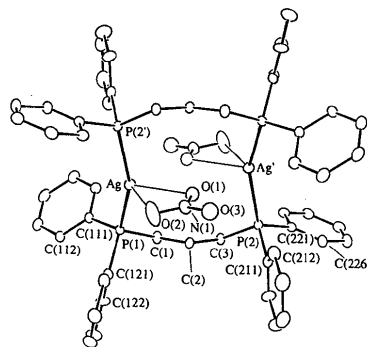

Fig. 1. Molecular structure and crystallographic numbering scheme employed for [Ag{Ph₂P(CH₂)₃PPh₂}NO₃]₂ (Johnson, 1971).

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Structure of a Nucleoside Analogue, 3'-Deoxy-2'-fluorothymidine*

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Abstract. 1-(2,3-Dideoxy-2-fluoro-β-D-*erythro*-pentofuranosyl)thymine, $C_{10}H_{13}FN_2O_4$, $M_r = 244.22$, monoclinic, $P2_1$, $a = 10.6001$ (7), $b = 6.1847$ (1), $c = 16.9227$ (4) Å, $\beta = 98.040$ (3)°, $V = 1098.52$ (8) Å³, $Z = 4$, $D_m = 1.47$, $D_x = 1.477$ Mg m⁻³, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 1.040$ mm⁻¹, $F(000) = 512$, $T = 298$ K, final $R = 0.033$ for 1868 unique observed reflections. The asymmetric unit contains two molecules (A and B). For molecule A: the N-glycosidic torsion angle χ has a value of -155.2 (2)° in the *anti* range; the sugar pucker is $^3T_2$ with $P = 12$ (1)° and $\psi_m = 35$ (1)° and the C4'—C5' conformation is $+sc$ with $\gamma = 55.1$ (3)°. For molecule B: the N-glycosidic torsion angle χ has a value

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* Structural Studies of Modified Nucleosides. Part III. Part II: Everaert, Peeters, Blaton & De Ranter (1990).

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